REMOVAL OF LEAD FROM AQUEOUS SOLUTION BY ADSORPTION ON COCONUT COIR ACTIVATED CARBON

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by

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Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Civil Engineering Department
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Approved by:

.....
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Project Supervisor

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

June 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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(SITI NURSHAMSHINAZZATULBALQISH BT SAMINAL)

ABSTRACT

Activated carbon was prepared from coconut coir and adsorption of lead from aqueous solution by the activated carbon was examined. Batch adsorption tests showed that extent of lead adsorption was dependent on metal concentration, contact time, pH and carbon dose. Adsorption was low at acidic pH and increased with pH. Equilibrium adsorption was attained in 150 min and maximum adsorption occurred at pH 5.0. Adsorption of lead by activated carbon followed pseudo-second-order kinetics. Adsorption capacity of the activated carbon for adsorption of lead was evaluated by batch equilibrium test and compared with that of F-400 commercial activated carbon. Coconut coir activated carbon showed higher adsorption capacity for lead [3.63 (Freundlich) and 7.75 (Langmuir)] than that [1.87 (Freundlich) and 7.55 (Langmuir)] of the F-400 activated carbon. Coconut coir activated carbon is a suitable substitute for commercial activated carbon in removal of lead from aqueous solution.

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LIST OF ABBREVIATION AND NOMENCLATURE

ABBREVIATION

PPE	Personal Protective Equipment
USA	United State of America
AAS	Atomic Adsorption Spectrophotometer
CCAC	Coconut Coir Activated Carbon
SEM	Scanning Electron Micrograph
VPSEM	Variable Pressure Scanning Electron Micrograph

NOMENCLATURE

KOH	Potassium Hydroxide
HCl	Hydrochloric Acid
NaOH	Sodium Hydroxide

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Industrial used water is one of the major sources of aquatic pollution. Among the aquatic pollutants, heavy metals have gained relatively more significance in view of their persistence, bio-magnification and toxicity. Lead is one of the ubiquitous and hazardous environmental pollutants where act as a cumulative poison and concentrates primarily in the bones. Inorganic lead (Pb^{2+}) is a general metabolic poison and enzyme inhibitor.

Treatment processes for lead removal from wastewater include precipitation, membrane filtration, ion exchange and adsorption (Kadirvelu *et al.*, 2001). Cost-effective alternative technologies or adsorbents for treatment of metal-containing wastewaters are needed. Due to their low cost, after these materials have been expended, they can be disposed of without regeneration. Generally, adsorbents can be assumed as low cost if they require little processing, are abundant in nature, or are a by-product or waste material from other industry.

Malaysia produces at least 120 millions coconuts annually. Coconut coir, therefore, is a waste by-product available at a minimal cost. The coconut husk fiber consisting of cellulose, hemicellulose, lignin and other functional groups, such as carbonyl, carboxyl, and hydroxyl, which are thought to have the potential to bind metal ions (Quek *et al.*, 1998).

1.2 Problem Statement

The removal of lead from contaminated water has become a major research topic due to toxicological problems caused by the toxic metals to the environment and to human health in recent years. Among various methods, adsorption has been proven to be an efficient technology. However, the application is limited due to high cost of the adsorbents. This research is conducted to investigate the feasibility of activated carbon prepared from coconut coir, as adsorbent of lead removal from aqueous solution.

1.3 Objective of Study

Three main objectives are as stated below:

i) To prepare activated carbon from coconut coir

ii) To determine the efficiency of coconut coir activated carbon to remove lead from water

iii) To compare effectiveness (adsorption capacity) of prepared coconut coir activated carbon with that of commercial activated carbon

1.4 Scope of Study

This project is in the form of laboratory experiments where coconut coir activated carbon (CCAC) was prepared to obtain absorbents with particles size range between 0.2 to 0.5mm. Stock solutions were prepared from lead nitrate and were diluted to get desired solutions to be used in adsorption experiments.

The aim of the present work is to investigate the removal of lead from aqueous solution using adsorption of CCAC by conducting two experiments, batch studies and adsorption isotherm studies.

In batch studies, the influence of experimental variables will be evaluated, such as contact time, solution concentration and adsorbent dose. Currently the affect of pH has been investigated. The purpose of varying the experimental variables is to determine the optimum parameters to get optimum lead removal from aqueous solution:

i) **Effect of solution pH**

Lead adsorption efficiency was observed by varying the pH value of lead solution. The selection of the optimum pH must be taken into account the fact that, precipitation of lead would occur if pH value selected is too high (Quek *et al.*, 1998).

ii) **Effect of initial concentration and contact time**

The selection of initial concentration and contact time is to maximise the adsorption efficiency.

iii) **Effect of adsorbent dose**

Adsorbent dose must be chosen correctly to obtain the desired adsorptive capacity.

The concentration of lead ions was determined by atomic absorption spectrophotometer (AAS). In adsorption isotherm studies, the parameters in batch studies will be used in the experimental work and isotherm data will be fitted to Freundlich and Langmuir models. Isotherm studies results will then be compared between commercial activated carbon and CCAC.

1.5 Relevancy of Project

Adsorbent obtained from agricultural waste, known as natural sorbent will be used instead of throwing away and increase the waste in country. The recycling opportunities of this waste can be developed. Furthermore, the other alternative as adsorbent for adsorption such as coconut coir can replace the expensive commercial activated carbon in the market, because the adsorbent is easily available and not bulky to handle. Low in density and high volatile content in coconut coir has made it suitable to produce reasonably high quality and low density of activated carbon.

1.6 Feasibility of Project

The feasibility study of the project within the scope and time frame is to get the best way how to manage the entire task in completing the research project. For this part of project, study and detailed research on the basic idea of the project has been done. All relevant information has been gathered for further guidance and reference in completing this study. Preparation of coconut coir activated carbon and preparation of lead solution has been done. Proper planning and procedures are developed to ensure a smooth project flow as well as to accomplish the project's objectives within the time period. The timeline of the study is shown in Figure 3.2 and Figure 3.3, Gantt charts.

CHAPTER 2
LITERATURE REVIEW

Review for the study was taken from journals and the internet. Basically, spot to be highlighted for the study consists of theory of adsorption and other type of lead removal practices and the limitations.

2.1 Adsorption

2.1.1 Adsorption Theory

Adsorption is the assimilation of a gas, liquid or dissolved substance by the surface of a solid and is physicochemical wastewater treatments process that gaining prominence as a means of producing high quality effluent that is low in dissolved organic compounds. Such waste streams require treatment; conventional methods for the removal of heavy metals from wastewater. Adsorption has become a well-established separation technique to remove dilute pollutants as well as offering the potential of regeneration, recovery and recycling of the adsorbed materials (Noroozi et al., 2008). The adsorption process is different with absorption process. Adsorption is the process by which molecules of a substance, such as a gas or a liquid, collect on the surface of another substance, such as solid. Meanwhile, absorption is the processes by which the molecules are attracted to the surface but do not enter the solid's minute spaces (Atkins, 1994).

Adsorption now is recognized as a significant phenomenon in most natural physical, biological and chemical processes. Sorption on solids, particularly active carbon, has become a widely used operation for purification and wastewaters. It plays an important role in many other processes of water treatment (Weber, 1972).

2.1.2 Adsorption Fundamentals

Adsorption on solids is a process which molecules in a fluid phase are concentrated by molecular attraction at the interface with a solid. The attraction arises from van der Waals forces, which are physical interaction between the electronic fields of molecule, and which also lead to such behaviour as condensation. Attraction to the surface is enhanced because the foreign molecules tend to satisfy an imbalance of forces on the atoms in the surface of a solid compared to atoms within the solid where they are surrounded by atoms of the same kind. The material adsorbing onto the solid phase is referred to as the adsorbate or adsorptive, the solid is called the adsorbent.

Adsorbents or activated carbon in particular, are typically characterized by highly porous structure. Adsorbents with the highest adsorption capacity for gasoline or fuel vapors have a large pore volume associated with pore diameters on the order of 50 Angstroms or less. When adsorption occurs in these pores, the process is comparable to condensation in which the pores become filled with liquid adsorbate. Adsorption process includes transfer of adsorbate molecules through the bulk phase to the surface of the solid, and diffusion onto internal surfaces of the adsorbent and into the pores (Burchell, 1999).

2.1.3 Adsorption by Activated Carbon

Adsorption is the process of accumulating substances that are in solution on a suitable interface. It also can define as a mass transfer operation in that a constituent in the liquid phase is transferred to the solid phase. The adsorption process has not been used extensively in wastewater treatment, but demands for a better quality of treated wastewater effluent, including toxicity reduction, have led to an extensive examination and use of the process of adsorption on activated carbon (Metcalf and Eddy, 2004).

The simplest way to conceptualize carbon adsorption is to think of the carbon particle as a porous ball. The water to be purified flows over the surface of, and between the balls. Solutes diffuse from the water stream into the pores of the ball and subsequently become adsorbed, or attached, to the surface of the pore like shown in Figure 2.1.

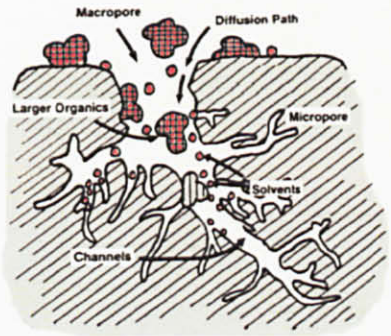

Figure 2.1: Solute absorbed to the surface of pores

Consequently, the rate of solute adsorption depends on how quickly a contaminant molecule can move from the bulk water flow to an unoccupied site within the balls' porous structures. The rate of adsorption is influenced by the number and size of the pores and temperature, but not the velocity with which the water flows over and between the balls.

The ideal particle would be small with many pores in order to maximise the distance the solute has to travel from the bulk fluid to an adsorption site. However, very small particles are difficult to contain and cause large pressure drops across the carbon bed. Solutes move more slowly at low temperatures, so that the rate of adsorption decreases as the temperature of the water decreases.

As the water enters a fresh carbon bed, contaminating solutes are swept to the surface of the initial layer of porous ball. Some of the solutes find their way into the porous structure and are adsorbed. The partially purified water flows to succeeding layers, where the process is repeated, and on through the bed.

As the porous ball at the inlet of the bed become saturated with solute, the incoming water will not be purified until it encounters a subsequent layer, where the porous balls still have adsorptive capacity. With the flow rates normally used through carbon beds, solutes are delivered to the surface of the balls much more quickly than they can be adsorbed.

As a result, some solutes may appear in the product stream, although at reduced concentrations, even though the porous ball may still have capacity to adsorb more solute.

Activated carbon is the most popular absorbent used in wastewater treatment. Several equilibrium studies on the adsorption of heavy metals using activated carbon and its derivatives have been carried out. Over the last decade, the low cost and commercial availability of biosorbents have stimulated great attention into the possibility of using various low cost adsorbents (Noroozi *et al.*, 2008)

2.1.4 Adsorption Isotherm

Proper analysis and design of adsorption separation process requires relevant adsorption equilibria as one of the vital information. In equilibrium, a certain relationship prevails between solute concentration in solution and adsorbed state (i.e., the amount of solute adsorbed per unit mass of adsorbent). Their equilibrium concentrations are function of temperature. Therefore, the adsorption equilibrium relationship at a given temperature is referred as adsorption isotherm (Febrianto *et al.*, 2009).

Equilibrium adsorption isotherms play an important role in the predictive modelling that is used for the analysis and design of adsorption systems. As such models cam be used to predict the performance of an adsorption process under a range of operating conditions, adsorption isotherms are invaluable tolls for theoretical evaluation.

However, no single model has been found to be generally applicable because although one isotherm equation may fit experimental data accurately under one set of conditions, it may fail entirely under another (Noroozi *et al.*, 2008).

Two most common isotherms which used to describe experimental isotherm data are Freundlich and Langmuir isotherm. Freundlich isotherm is the most commonly used isotherm to describe the adsorption characteristic of activated carbon used in water and wastewater treatment (Tchobanoglous *et al.*, 2004).

The Freundlich isotherm is capable of describing the adsorption of organic and inorganic compounds on a wide variety of adsorbents (Febrianto *et al.*, 2009). The Freundlich equation has the general form (Weber, 1972):

$$q_e = K_F C_e^{1/n}$$

The Freundlich equation is basically empirical but is often useful as a means for data description. Data are usually fitted to the logarithmic form of the equation (Weber, 1972):

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$

where; q_e = amount of adsorbate adsorbed per unit weight of adsorbent, mg adsorbent per g of activated carbon (mg/g)

K_F = adsorption capacity of activated carbon, (mg adsorbate/ g activated carbon) (L water/ mg adsorbate)$^{1/n}$

C_e = equilibrium concentration of adsorbate in solution after adsorption, mg/L

1/n = adsorption intensity of activated carbon

The plot of log q_e versus log C_e has a slope with the value of $1/n$ and an intercept magnitude of log K_F. log K_F is equivalent to log (x/m) when C_e equals to unity. However, in other case when $1/n \neq 1$, the K_F value depends on the units upon which q_e and C_e are expressed. On average, a favourable adsorption tends to have

Freundlich constant n between 1 to 10. Larger value of n (smaller value of $1/n$) implies stronger interaction between adsorbent and heavy metal while $1/n = 1$ indicates linear adsorption leading to identical adsorption energies for all sites (Site, 2001)

The Langmuir adsorption model is valid for single-layer adsorption. The Langmuir adsorption isotherm is:

$$q_e = (Q^\circ bC_e) / (1 + bC_e)$$

where; Q° = number of moles of solute adsorbed per unit weight of adsorbent in forming a complete monolayer on the surface

q_e = number of moles solute adsorbed per unit weight at concentration C_e

b = constant related to the energy or net enthalphy, ΔH, of adsorption.

Although the basic assumptions explicit in development of the Langmuuir isotherm are not met in most adsorption system of concern for water and wastewater treatment, the Langmuir isotherm has been found particularly useful for description of equilibrium data for such systems and for providing parameters (Q° and b) with which to quantitatively compare adsorption behaviour in different adsorbate-adsorbent systems. Rarely does a value of Q° developed for adsorption on activated carbon represent a true monolayer capacity, but it does represent a practical limiting capacity for adsorption (Weber, 1972).

Adsorption isotherm of lead adsorption by coconut coir activated carbon and commercial activated carbon will be fitted to the linear form of the Langmuir adsorption isotherm:

$$C_e / q_e = (1 / b Q^\circ) + (C_e / Q^\circ)$$

The Langmuir constants Q° and b for lead adsorption will be compared between prepared activated carbon and commercial activated carbon for which shows the higher limiting capacity of adsorption.

2.1.5 Adsorption Kinetics

Kinetic studies in adsorption of heavy metals are important to determine the efficiency of adsorption. It is also necessary to identify the adsorption mechanism type in a given system. On the purpose of investigating the mechanism of adsorption and its potential rate-controlling steps that include mass transport and chemical reaction processes, kinetic models have been exploited to test the experimental data. Information on the kinetics of metal uptake is required to select the optimum condition for full-scale batch metal removal processes.

Adsorption kinetics is expressed as the solute removal rate that controls the residence time of the sorbate in the solid-solution interface. In practice, kinetic studies were carried out in batch reactions and linear regression was used to determine the best-fitting kinetic rate equation. Several adsorption kinetic models have been established to understand the adsorption kinetics and rate-limiting step. These include pseudo-first and pseudo-second order rate model and Weber and Morris sorption kinetic model. The pseudo-first and pseudo-second order kinetic models are the most well-liked model to study the adsorption kinetics of heavy metals and quantify the extent of uptake in adsorption kinetics (Febrianto *et al.*, 2009).

The Lagergren Pseudo-first-order rate expression for heavy metal adsorption mechanism onto the adsorbent:

$$\log (q_e - q_t) = \log q_e - (k_1 t / 2.303)$$

Pseudo-second-order rate expression:

$$(t / q_t) = (1 / k_2 q_e^2) + (t / q_e)$$

where; q_t and q_e = adsorbed lead amount at time t

k_1 and k_2 = rate constant

By linear plots of value $log\ [(q_e - q_t) / q_e]$ against time t will give the pseudo-first-order adsorption rate constant k_1 from the slopes and q_e can be calculated from the intercept.

Similarly, by plotting t/q_t against time t, the pseudo-second-order adsorption rate constant k_2 and q_e can be determined from slope and intercept of plot (Liu and Zhang, 2009).

Mathematical model described by Weber and Morris commonly used to provide the definite adsorption mechanism (Weber and Morris, 1963):

$$q_t = K_d t^{1/2} + C$$

where: K_d = intra-particle diffusion rate constant

C = adsorption constant

q_t = adsorbed lead amount at time t

The results of linearity test of q_t against $t^{1/2}$ will shows K_d and C obtained from plot slopes and intercepts. Higher the slopes implied that heavy metal ions transferred from bulk solution to adsorbent surface is faster. Meanwhile, lower slopes suggest that intra-particle diffusion is rate-controlling step after a long contact time (Liu and Zhang, 2009).

2.2 Adsorbent

2.2.1 Definition

The material being concentrated adsorbed in the adsorbate is termed as adsorbent. The more a substance likes the solvent system, the more hydrophilic in the case of an aqueous solution, the less likely it is to move toward an interface to be adsorbed.

Conversely, a hydrophobic (water disliking) substance will more likely be adsorbed form aqueous solution (Weber, 1972).

Adsorbent is a solid, liquid or gas onto which the adsorbate accumulates. Adsorbent is capability of adsorption and having the capacity or tendency to adsorb. The principle types of adsorbents include activated carbon, synthetic polymetric and silica-based adsorbent.

2.2.2 Coconut Coir

Coir is a coarse fibre extracted from fibrous outer shell of a coconut. Coir fibres are found between the hard, internal shell and the outer coat of a coconut. The individual fibre cells are narrow and hollow, with thick walls made of cellulose. They are pale when immature but later become hardened and yellowed as a layer of lignin is deposited on their walls. There are two varieties of coir. Brown coir is harvested from fully ripened coconuts. It is thick, strong and has high abrasion resistance. It is typically used in mats, brushes and sacking. Mature brown coir fibres contain more lignin and less cellulose than fibres such as flax and cotton and so are stronger but less flexible. White coir fibres are harvested from the coconuts before they are ripe. These fibres are white or light brown in color and are smoother and finer, but also weaker. The coir fibre is relatively water-proof and is one of the few natural fibres resistant to damage by salt water.

2.2.3 Activated Carbon

Activated carbon is a form of carbon that has been processed to make it extremely porous thus to have a very large surface area available for adsorption or chemical reactions. Activated carbon is prepared by the pyrolysis of a variety of organic materials (e.g. coconut shells, bones, coal, lignite, peat and wood) in closely regulated atmospheres. The porosity of activated carbon and, thus, its internal surface area,

varies with the type of material used and the conditions of pyrolysis. The presence of oxygen alters the surface of the carbon inside the porous structure, changing its adsorptive properties.

Depending on the material from which the carbon is prepared, various amounts and types of residual metallic contaminants, including aluminium, may be left in the activated carbon. These contaminants may have the potential to leach into water as it flows through the carbon.

Adsorbents combine chemical and physical processes to remove organic contaminants and the compound that import color, taste, and odor to water. The most commonly-used adsorbent is activated carbon – a substance which is quite similar to common charcoal. Activated carbon however, is treated by heat and oxidation so that it becomes porous and able to readily adsorb, or capture the impurities found in water.

Activated carbon also attracts not only known contaminants, but also naturally dissolved organic matter (much of which is harmless). Therefore, monitoring is needed to ensure the carbon doses are high enough to adsorb all contaminants. There are two different forms of activated carbon in common use, granular activated carbon (GAC) and powdered activated carbon (PAC). Physically, the two differs by particle size and diameter. Overall, activated carbon is better than ion exchange for removing organic substances.

2.2.4 Application in Industries

Activated carbon is used in gas purification, metal extraction, water purification, medicine, sewage treatment, air filters in gas masks and filter masks, filters in compressed air and many other application. Carbon adsorption has numerous applications in removing pollutants from air or water streams both in the field ad in

industrial approach such as groundwater remediation and drinking water purification. In water treatments, special attention is given to removal of dangerous inorganic material such as the heavy metals, including mercury, chromium, copper, zinc, cobalt, nickel, lead, cadmium, arsenic and iron.

The activated industry, like any other industry, survives only on the profits it makes. The common strategy is to buy lower and sell higher. Activated carbons are not high-value products and so profit margins need to be carefully regulated. Therefore, the industry need to occupy stable, abundant supplies of raw (parent) materials, from nearby source to keep the transportation costs to a minimum, and free from the cycles of cropping (Harry Marsh, 2006).

Figure 2.2: Coir bricks or bedding

As for coconut coir, here are convenient Coir Bricks or Bedding in the market as shown in Figure 2.2. The grow coir is a natural fibre made from coconut husks. However, coconut coir is already known for a few applications such as mat and mattress product, medium for hydroponics and container plant growing for outdoor gardening. Therefore, this experiment will discover new benefit of coconut coir as activated carbon for lead removal.

2.2.5 Manufacturing of Activated Carbon

Adsorbent obtained from agricultural waste, known as natural sorbent will be used instead of throwing away and increase the waste in country. The recycling opportunities of this waste can be develop. Furthermore, the other alternative as adsorbent for adsorption such as coconut coir can replace the expensive commercial activated carbon in the market, because the adsorbent is easily available and not bulky to handle.

Nowadays, the heavy metal removal from the effluent water has been one of the concerns during the wastewater treatment process. To overcome any adverse effect cause from releasing of the metal waste, activated carbon prepared from coconut coir is studied by investigating its efficiency to adsorb lead from aqueous solution.

There are few efficient sorbents from agricultural waste and natural sorbent such as crab shell (Lee *et al.,* 1998), pine wood and rice husk (Liu and Zhang, 2009), coir pith (Santhy and Selvapathy, 2006), hazelnut husk (Imamoglu and Tekir, 2008), cashew nut shells (Tangjuank *et al.*, 2009), periwinkle shells (Badmus *et al.*, 2007), peanut shell (Xu and Liu, 2008), coconut shells (Gimba *et al.*, 2009) and bamboo dust (Kannan and Veemaraj, 2009) have been used. Still, metal contaminated wastewater treatment needs new adsorbents that are economic, easily available and effective. Before removing lead from aqueous solution, activated carbon must be prepared from the natural sorbent by chemical process (Santhy and Selvapathy, 2006)

2.2.6 Commercial Activated Carbon

Commercial activated carbon used in the experiment is F-400 activated carbon, obtained from Calgon Corporation, USA.

2.3 Adsorbate

2.3.1 Definition

Adsorbate is the substance that is adsorbed. Adsorbate is a material that has been or is capable of being adsorbed. The adsorbate is the substance that is being removed from the liquid phase at the interface (Metcalf and Eddy, 2004).

2.3.2 Lead [Pb(II)]

Lead is a soft, malleable poor metal, also considered to be one of the heavy metals. Lead has a bluish-white color when freshly cut, but tarnished to a dull-grayish color when exposed to air. It has a shiny chrome-silver luster when melted into a liquid. Elemental lead occurs in nature in form of ores. Once lead is mined, processed and introduced into environments, it is a potential problem.

Leads are used in building construction, lead acid batteries, bullets and shots, and are part of solder, pewter, fusible alloy and radiation shields. Lead is a poisonous metal that can damage nervous connections (especially in young children) and cause blood and brain disorders. Like mercury, lead is a potent neurotoxin that accumulates in soft tissues and bone over time.

Lead has been commonly used for thousand of years because it is widespread, easy to extract and easy to work with. In the early Bronze Age, lead was used with antimony and arsenic. Lead is usually found in ore together with zinc, silver and (most abundantly) copper, and is extracted together with these metals.

2.3.3 Application of Lead in Industries

Lead is a major constituent of the lead-acid batteries. It is used as a coloring element in ceramic glazes, as projectiles, in some candles to threat the wick. It is the traditional base metal for organ pipes, and is used as electrodes in the process of electrolysis. One of its major uses is in the glass of computer and television screens, where it shields the viewer from radiation. Other uses are in sheeting, cables, solders, lead crystal glassware, ammunitions, and bearings and as weight in sport equipment.

2.3.4 Environmental Effects of Lead

Lead may be present in the waste stream from the industries that uses lead or it may also contaminate drinking water through corrosion of pipes. This is more likely to happen where the water is slightly acidic.

Lead can cause unwanted effects such as anaemia, rise in blood pressure, kidney damage, miscarriage and subtle abortion, disruption in nervous system, brain damage, declined fertility of men through damage of sperm, diminishing learning abilities of children, and behavioural disruption of children such as aggression, impulsive behaviour and hyperactivity.

Lead accumulates in the bodies of water organisms and soil organisms and affects the whole food chain.

2.4 Lead Removal by Activated Carbon

The presence of lead in the environment is of major concern because of their toxicity and threat to human life and environment. Anthropogenic sources of heavy metals include wastes from the electroplating and finishing industries, metallurgical industry, tannery operations, chemical manufacturing, mine drainage, battery manufacturing, and leachates from landfills and contaminated groundwater from hazardous waste sites. For wastes with high metal concentrations, precipitation processes (e.g. hydroxide, sulphide) are often most economical. However, given the tendency for stricter heavy metal limits, additional treatment processes, down line from the precipitation process, may be required to "polish" the effluent prior to discharge. These tertiary processes may also be the primary metal removal process for waste streams with low concentration of metals for drinking water. Examples of tertiary processes include ion exchange, reverse osmosis and adsorption. Ion exchange and reverse osmosis while effective in producing an effluent low in metals, have high operation and maintenance cost and are subjected to fouling (Reed, 2002).

2.4.1 Carbon Surface Chemistry

The type and number of activated carbon surface group will influence the extent and rate at which organic and inorganic compound are adsorbed. It is not known what surface functional groups are formed during the carbon activation process. Whether the surface oxides are acidic, basic or amphoteric in nature depends on the method of activation. Carbons capable of adsorbing strong base are called L-type (acidic carbons) and those capable of adsorbing strong acid are H-type (basic carbons). L-type carbons are generally exposed to oxygen at temperatures of 200-500C or solution oxidants during the activation process. H-type carbons are formed using activation methods that remove indigenous surface oxide groups. This is accomplished by heating the carbon in the presence of an inert gas or vacuum and

cooling to low temperatures in a similar environment. Thus, H-type carbons are exposed to oxygen only at low temperatures, and the oxidizing conditions necessary for acidic functional group to form are minimized (Reed, 2002).

2.4.2 Heavy Metal Removal Mechanisms

Heavy metal can be removed by the following phenomena: physical adsorption, chemisorption, hydrogen bonding, ion exchange, surface precipitation and filtration. Physical adsorption in non-specific in nature and is recognized as the primary removal mechanism for organic adsorbates. Chemisorption is specific in nature and involves the formation of a covalent bond (sharing of electrons) between the adsorbate and a specific carbon surface site. Chemisorption is generally considered to be irreversible, while physical adsorption is considered to be reversible. In hydrogen bonding, a long-range attractive force exists between the hydrogen atom of hydrated metal ions and carbon surface oxygen groups. Hydrogen bonding can be classified under chemisorption, but the hydrogen bond is much weaker than a covalent bond. With covalent bonds, the much stronger inner-sphere complex is formed, while an outer-sphere complex is formed for hydrogen bond. Ion exchange is the sorption phenomenon that occurs when the adsorbate and adsorbent are oppositely charged. Precipitation of a metal on surface is easier than the formation of the same solid in solution. For this reason, given the proper chemical conditions, heavy metal will preferentially precipitate on carbon surface. Localized high concentrations of the metal and OH^- in an activated carbon's extensive pore volume can enhance metal removal (Reed, 2002).

2.5 Lead Removal Processes

There are a few techniques commonly used in the current practice:

1) Hydroxide Precipitation

   Limitations associated with hydroxide treatment include:

   a) Hydroxide precipitation tend to solubilize if the solution pH is changed
   b) Removal of metal by hydroxide precipitation in mixed metal wastes may not be effective since the minimum solubility for different metal occurs at different pH values.(Esalah *et al.*, 1999)

2) Carbonate Precipitation

   Although carbonate precipitates have better filtration characteristics than hydroxide precipitate, carbonate precipitation did not reduce the residual concentration below that obtained by hydroxide precipitation. (Esalah *et al.*, 1999)

3) Sulphide Precipitation

   Sulphide precipitation is an effective alternative to hydroxide precipitation for removing heavy metals. A high degree of separation was obtained by precipitation with sodium sulphide to remove lead, cadmium and zinc from wastewater.

   However, the sulphide treatment may generate toxicity problems from excess sulphide reagent. Thus, the removal of excess sulphide requires further treatment (Esalah *et al.*, 1999).

4) Natural Phosphate Adsorption

Natural phosphate exhibits significant catalytic activity which is closely related to adsorption. The actions of solid catalysts result from their ability to adsorb reacting substances. After the performance in catalysis, the calcined phosphate showed a strong ability for removal of Pb^{2+}, Cu^{2+} and Zn^{2+} from aqueous solution (Mouflih *et al.*, 2005).

5) Crab Shell Particles Adsorption

Crab shell particles, mainly composed of calcium carbonate and chitin, took up lead from aqueous solutions by dissolution of calcium carbonate followed by precipitation of lead. The micro precipitates formed were then adsorbed to the chitin on the surface of crab shell particles. (Lee *et al.*, 1998)

6) Coir Pith Activated Carbon Adsorption

The activated carbon prepared from coconut coir pith by potassium hydroxide activation was found to exhibit remarkable adsorption capacity for cadmium, copper and zinc. (Santhy and Selvapathy, 2004)

7) Biochars Adsorption

Hydrothermal conversion of biomass into biofuel could produce a special type of biochar as byproduct. Biochars prepared from hydrothermal liquefaction of pinewood (P300) and rice husk (R300) were found containing large amount of oxygen which were quite effective for lead removal. (Liu and Zhang, 2009)

CHAPTER 3
METHODOLOGY

3.0 Chemicals/ Reagents

1) Lead Nitrate
2) Hydrochloric Acid
3) Potassium Hydroxide

3.1 Tools/ Equipment

1) Atomic Adsorption Spectrophotometer (AAS)
2) Mortar and Pestle

3.2 Lead Solution Preparation

100 mg/L concentration of lead solution (stock solution) was prepared by weighting 159.85 mg lead (II) nitrate [$Pb(NO_3)_2$]. Before preparing stock solution in a 1000 mL volumetric flask, it was being washed using nitric acid to avoid adsorption of lead by the glass wall. The weighed lead (II) nitrate were then dissolved using distilled water. Desired solutions for further experiments were prepared by dilution of 1 L stock solution.

3.3 Coconut Coir Activated Carbon Preparation

Raw coconut coir was outsourced from a nursery in Taman Maju, Sri Iskandar. Coconut coir activated carbon was prepared according to a method used by Santhy and Selvapathy (2004) to prepare coir pith activated carbon.

Figure 3.1: Raw coconut coir

Coconut coir was sieved and washed several times with distilled water and later dried at 110°C for 24 hr. Dried coconut coir was treated with 10% potassium hydroxide (KOH) solution and kept overnight at 105°C. The dried treated coconut coir was then be subjected to activation at 900°C for 30 min in an atmosphere of nitrogen. The carbon obtained was washed again with distilled water and then with 10% hydrochloric acid. The carbon was washed again with distilled water to remove the free acid and dried at 110°C for 24 hr. The activated carbon was ground to a finer size using mortar and pestle to a particle size of 0.2-0.5mm.

3.4 Batch Study Using Coconut Coir Activated Carbon

Studies were carried out by adding known amount of coconut coir activated carbon to a series of flask containing 25ml of solution prepared earlier. The solutions were equilibrated in a rotary mechanical shaker at 150 rpm and 25°C. After a certain desired time, the aliquots were filtered through Whatman No.1 filter paper.

The amount of metal ion adsorbed was computed as:

$$\text{Adsorbate uptake (\%)} = \frac{(C_0 - C_t) \times 100}{C_0}$$

where C_0 and C_t are the initial and at time (t) of the adsorbate concentration, respectively.

The influence of experimental variables, such as contact time, initial solution concentration, pH and carbon dose will be evaluated. The pH of solution was adjusted using sodium hydroxide (NaOH) or hydrochloric acid (HCl). The concentration of lead was determined by atomic adsorption spectrophotometer method.

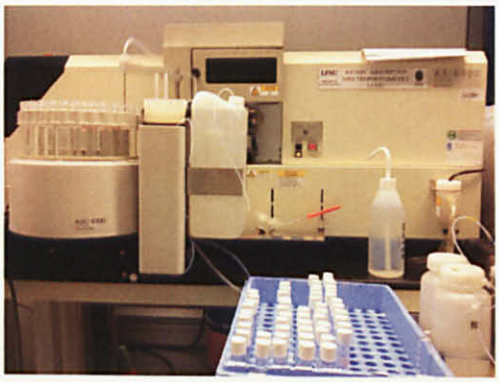

Figure 3.2: Atomic adsorption spectrophotometer (AAS)

3.4.1 Effect of pH

Experiment was carried out by varying the pH value at 24hr contact time. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to adjust the initial pH ranging from pH 1-6. A series of 25 mL of 20 mg/L initial concentration of lead solution were taken and adjusted to different initial pH value. 0.1 g of coconut coir carbon were added into the mixture and were equilibrated in a 150 rpm orbital shaker at room temperature for 24hr. The reading for each solution was taken after 24hr contact time, filtering through Whatman No.1 filter paper. The final lead solution concentration was determined using AAS and amount of lead absorbed and percentage removal was calculated.

3.4.2 Effect of Initial Concentration and Contact Time

25 mL of different initial concentration (20 mg/L, 40 mg/L and 60 mg/L) at optimum pH each lead solution were taken in series of 125 mL conical flasks containing 0.1 g of powdered coconut coir carbon (200-500 μm particle size). The flasks were equilibrated using 150 rpm orbital shaker at 25°C for a pre-determined time. The progress of adsorption during the experiment was determined by taking the aliquots after desired contact time (1 hr-3 hr), filtering through Whatman No. 1 filter paper. The supernatant was analyzed using AAS for each lead concentration, 20 mg/L, 40 mg/L and 60 mg/L. The amount of lead adsorbed was then calculated.

3.4.3 Effect of Adsorbent Dose

Effect of adsorbent dose was studied by varying amount of powdered coconut coir activated carbon weight from 0.1 g to 0.4 g in 25 mL at optimum contact time and pH. The desired concentration of lead solution was prepared from stock solution and 25 mL of solution was taken in a series of 125 mL conical flasks where 0.1 g, 0.2 g,

0.3 g and 0.4 g of carbon were added into each flask, respectively. The flasks were then equilibrated at 150 rpm in an orbital shaker, for the optimum contact time. Aliquots were filtered through Whatman No.1 filter paper and lead concentration were measured by AAS.

3.5 Adsorption Kinetic Study

In all experiments, a known amount of adsorbent was thoroughly mixed with 1000 mL solution with pre-determined initial concentration. Immediately after adding the adsorbent, the mixture was shaken on an orbital shaker at 150 rpm at 25°C. 2 mL samples were removed after a required contact time, which varied between 5 min and optimum contact time, and filtered through Whatman No.1 filter paper. Filtrate was analysed for lead ion concentration by AAS.

3.6 Adsorption Isotherm Study

The concentration of lead solution was varied by ten different concentrations (10 mg/L to 100 mg/L). All the solutions were adjusted to pH optimum determined in batch study. The series of 25 mL of different concentrations were equilibrated for optimum contact time after adding 0.1 g of coconut coir activated carbon into the solution. The flasks were equilibrated at 150 rpm in an orbital shaker. The aliquots were taken after the optimum contact time, filtering through Whatman No.1 filter paper. The lead solutions filtrate was measured by AAS. The same experiment was done using the F-400 activated carbon.

The experimental data were analysed by fitting to Freundlich and Langmuir adsorption isotherms. For the Freundlich isotherm, the value of K_F (y-intercept) and $1/n$ (slope) which indicate the adsorption capacity and adsorption intensity of coconut coir carbon were compared to that of commercial activated carbon. For the Langmuir

isotherm, the value of Q° and b which indicate the adsorption capacity and adsorption energy of coconut coir carbon, were compared to that of F-400 activated carbon.

3.7 Hazard Analysis

Hazard analysis is a systematic process and proactive approach for identifying hazards and recommending corrective actions. Hazard sources can be classified into sources of motion, sources of extreme temperatures, types of chemical exposures, sources of harmful dust, sources of light radiation, sources of sharp objects and any electrical hazards.

In this study, chemical laboratory will become the workplace to prepare the absorbent, adsorbate and for the adsorption process. Working in hot conditions can cause health effects ranging from discomfort to serious illness. Therefore, identify the information on heat, temperature and pressure hazard at laboratory to overcome the hazard associated with the extreme heat, temperature and pressure (110°C - 900°C). In this study, hot usage of furnace, fixed bed activation unit in Figure 3.3 and oven will be exposed. To overcome any hazard happen, wearing gloves when taking out the sample from oven can protect direct heat from the source. The operation must be under laboratory technician supervision.

Figure 3.3: Fixed bed activation unit

Second hazard is mechanical hazard, especially while using cutter, blender or grinder to grind the coconut coir. Body parts are in contact with sharp edges. This hazard will occur at the point of cutting operation. Safeguards are essential for the protection purposes and prevention from injuries. Gloves must be worn to avoid the sharp blade and the operation must be under laboratory technologist's supervision.

Chemical hazard can occur while handling the chemical in chemical process of preparation the activated carbon and pH studies. Concentrated acid or alkali is corrosive and must therefore be handled with appropriate care, since it can cause skin burns, permanent eye damage and irritation. Latex gloves offer no protection, so rubber glove, should be worn when handling the compound. Certain chemicals are very high of concentrated, handle it inside laboratory fume hood and avoid smelling the fume of the chemicals.

To prevent any accident happen and to ensure a safe work flow is practiced, student must attend the briefing session about laboratory in how to use or handle the tools, equipments or chemicals in proper ways. For the safety precaution, student should wear appropriate personal protective equipment, PPE and clothing such as lab coat and closed shoe while entering the laboratory area. Prevent human contact with any potentially harmful machine part and handle the chemical properly.

3.8 Data Collection and Planning

All data and information that are related to study will be obtained from experimental analysis and many references. Information from journals, websites on related topic and text books were gathered to achieve the objective of the research which is to investigate the efficiency of coconut coir activated carbon. Meanwhile, the project planning is shown on the Gantt chart.

Week No	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	semester break
Activities																					
Briefing Session										M											
Technical Writing Workshop										I								E			
IRC Workshop										D								X			
Lab Briefing Session																		A			
Journals										S								M			
Progress Report										E								I			
HSE Talk										M								N			
Referencing										E								A			
Interim Report										S								T			
Oral Presentation										T								I			
Initiative										E								O			
Topic and SV Confirmation										R								N			
Topic Understanding																					
Journals										B											
Methodology Understanding										R								W			
Experiment Scheduling										E								E			
Progress Report Discussion										A								E			
Coconut Coir Activation										K								K			
Experimental Work																					
FYP 2 Forecast																					

Figure 3.1: FYP I Gantt chart

Week No	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
Activities																				
Briefing Session								M												
Seminar I (IEM Talk)								I									E			
Statistical Analysis Talk								D									X			
Progress Report I & II																	A			
Poster Exhibition								S									M			
Softbound Dissertation Submission								E									I			
Oral Presentation								M									N			
Hardbound Dissertation Submission								E									A			
								S									T			
								T									I			
Initiative								E									O			
Coconut Coir Activation								R									N			
Experiment: Batch Study																				
Progress Report Discussion								B												
Experiment: Adsorption Isotherm Study								R									W			
Poster Preparation								E									E			
Data Collection and Discussion								A									E			
Dissertation Discussion and Preparation								K									K			
Oral Slides Preparation & Mock Presentation																				

Figure 3.2: FYP II Gantt chart

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Coconut Coir Activated Carbon Preparation

Lab experiment was conducted to prepare the stock lead solution for early exposure and familiarization. The raw coconut coir was treated with potassium hydroxide and subjected to the activation process. The prepared activated carbon is shown in Figure 4.3.

Figure 4.1: Raw coconut coir

Figure 4.2: Treated coconut coir

Figure 4.3: Coconut coir activated carbon

4.2 Physicochemical Characteristic of Activated Carbon

The tests were conducted to study the physicochemical characteristics of the prepared coconut coir activated carbon and F-400 activated carbon.

4.2.1 Scanning Electron Micrograph

Scanning electron micrograph (SEM) of coconut coir activated carbon and F-400 activated carbon were observed by Variable Pressure Scanning Electron Microscope (VPSEM) with different magnifications. The figures below show the SEM.

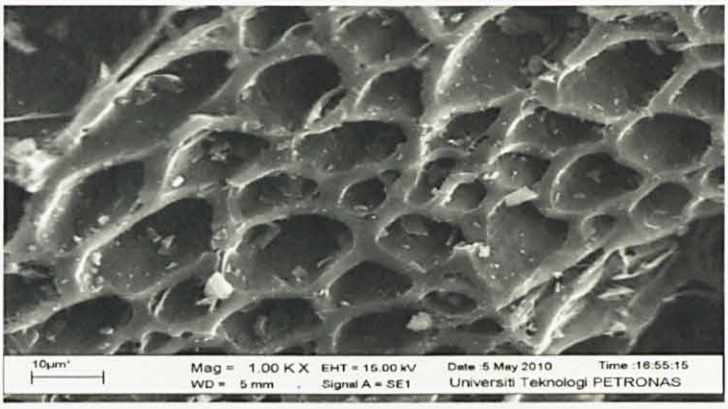

Figure 4.4: SEM micrograph of coconut coir activated carbon

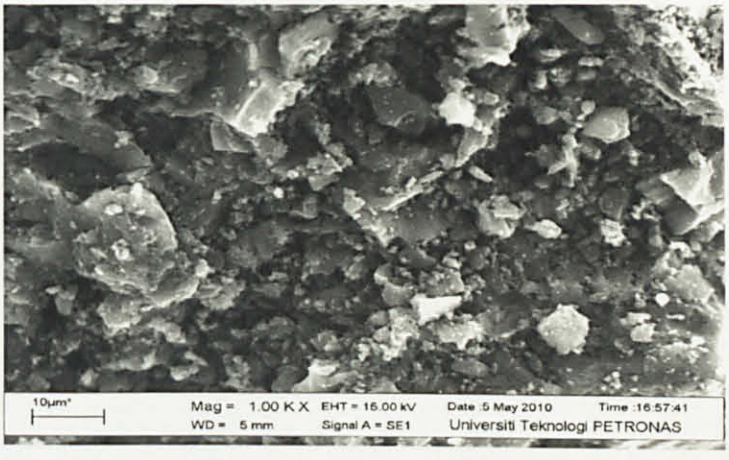

Figure 4.5: SEM micrograph of F-400 activated carbon

The SEM micrographs of coconut coir activated carbon in Figure 4.4 and F-400 activated carbon in Figure 4.5 were under 1000 magnification. Coconut coir shows a fibrous structure and circular pores distributed across its surface. The honeycomb-shaped pores indicated that coconut coir has a large surface area available for adsorption. F-400 activated carbon SEM image reveals rough, fragmented and uneven surface. This showed that F-400 activated carbon surface area is also considerably large.

4.3 Adsorption Study

4.3.1 Batch Study Using Coconut Coir Activated Carbon

The adsorption rates of lead onto coconut coir activated carbon in a batch adsorption system by varying the parameters have been studied through the experiments. The influence of experimental variables, such as contact time, solution concentration and carbon dose were calculated. The percentage of the adsorbate uptake was calculated by the following equation:

$$Adsorbate\ uptake\ (\%) = (C_o - C_t) / C_o \times 100$$

where, C_o and C_t are the initial and at time (T) of the adsorbent concentration, respectively.

4.3.1.1 Effect of pH

To determine the effect of pH, 0.1 g of coconut coir activated carbon was added to series of flasks containing 25 mL of 20 mg/L solutions and contact time used was 24hr. The pH of solutions was varied from pH 1.0 to pH 6.0. From the experimental results, in Table 4.1 shows that sorption is very low at acidic pH and increases with increasing pH of Pb(II) solution. The optimum lead removal was observed at pH 5.0.

Table 4.1: Effect of pH on adsorption of lead onto coconut coir activated carbon

pH	C_o	C_t	Ads (%)
1	21.8	18.66	14.40366972
1.5	21.8	18.01	17.3853211
2.5	21.8	16.13	26.00917431
3	21.8	15.08	30.82568807
4	21.8	13.47	38.21100917
5	21.8	13.28	39.08256881
6	21.8	13.15	39.67889908

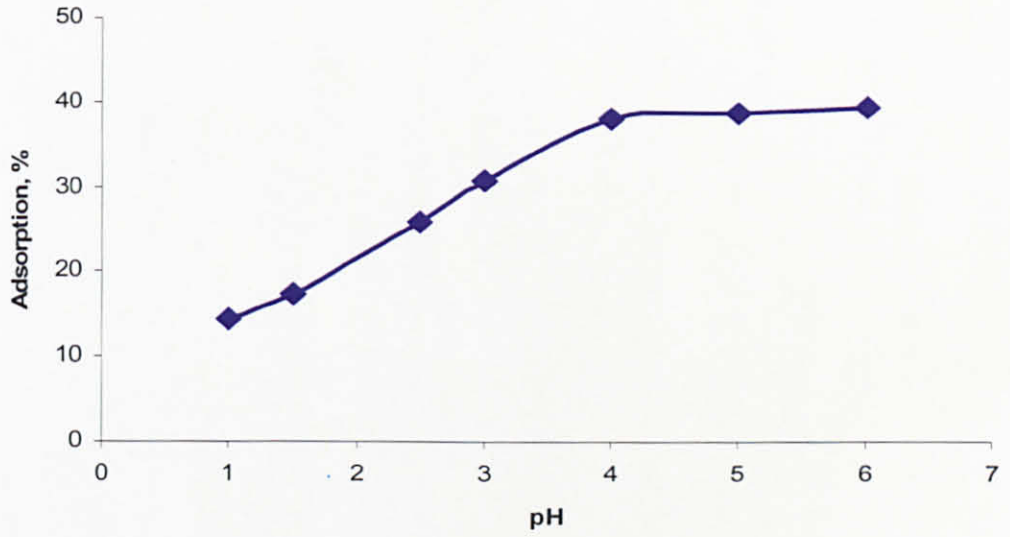

Figure 4.6: Effect of pH on adsorption of lead onto coconut coir activated carbon

The effect of pH on the removal of lead was summarized in Figure 4.6. From the graph, it was observed that optimum pH value was found at pH 5.0. From the Figure 4.6, it was evident that the adsorption is increasing with the increasing of pH.

This result showed similar trend to lead removal by pomegranate peel (El-Ashtoukhy *et al.* 2008); and by heartwood of areca catechu powder (Chakravarty *et al.* 2010).

4.3.1.2 Effect of Initial Concentration and Contact Time

To investigate the efficiency of coconut coir activated carbon, the effect of initial concentration and contact time on removal of lead is shown in Table 4.2 below. The contact time were varied from 0 min to 180 min for three different concentrations 20mg/L, 40 mg/L and 60 mg/L of lead solutions at pH 5 in an orbital shaker. Removal of lead was found to be complete at 150 min for all three concentrations.

Table 4.2: Effect of initial concentration and contact time on adsorption of lead onto coconut coir activated carbon

Time (min)	20 mg/L			40 mg/L			60 mg/L		
	Co	Ct	Ads (%)	Co	Ct	Ads (%)	Co	Ct	Ads (%)
0	19.98	19.69	1.4489	39.72	39.2982	1.06741	62.01	61.4439	0.91387
1	19.98	16.47	17.5650	39.72	34.0128	14.3733	62.01	53.6077	13.5507
10	19.98	14.34	28.2177	39.72	30.0707	24.2975	62.01	48.1061	22.4227
20	19.98	13.81	30.8618	39.72	28.8831	27.2872	62.01	47.381	23.5921
30	19.98	13.48	32.5090	39.72	28.1823	29.0515	62.01	45.7526	26.2181
45	19.98	12.81	35.8718	39.72	26.9532	32.1457	62.01	45.0394	27.3682
60	19.98	12.50	37.4284	39.72	26.1508	34.1657	62.01	43.8954	29.2130
90	19.98	12.38	38.0255	39.72	25.6119	35.5224	62.01	42.7146	31.1172
120	19.98	12.28	38.4985	39.72	25.4208	36.0035	62.01	42.1736	31.9896
150	19.98	12.18	39.0365	39.72	25.3941	36.0707	62.01	42.1648	32.0038
180	19.98	12.15	39.1531	39.72	25.1389	36.7132	62.01	41.8932	32.4418

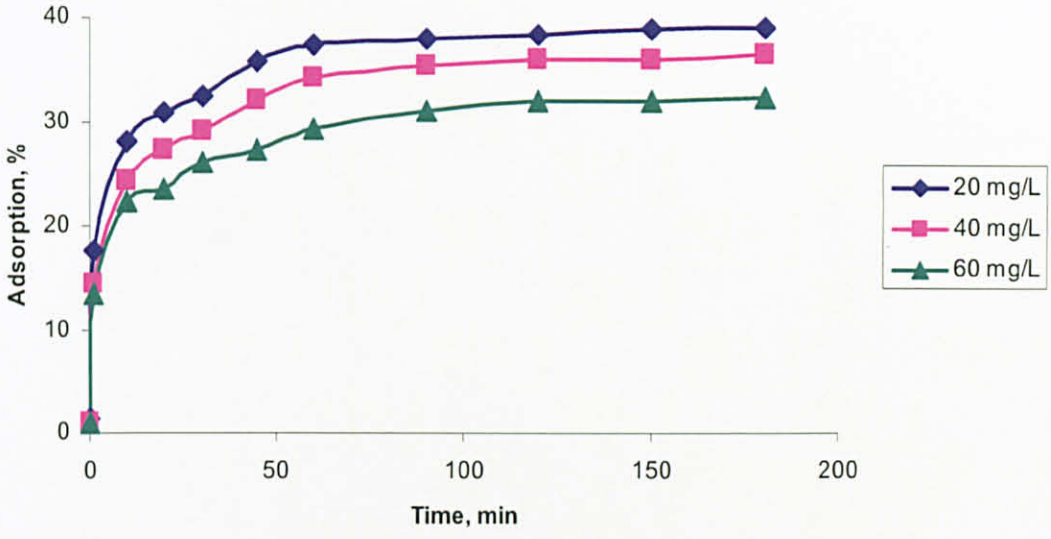

Figure 4.7: Effect of initial concentration and contact time on adsorption of lead onto coconut coir activated carbon.

Figure 4.7 shows the effect of initial concentration and contact time on the adsorption of lead. Adsorption percentage increased with the decrease in initial concentration of lead. Adsorption increased with the increase in contact time and reached equilibrium at 150 min for all three concentrations. This result showed similar trend to lead removal by silica ceramic (Salim and Munekage, 2009); and by coconut pith activated carbon (Santhy and Selvapathy, 2004).

4.3.1.3 Effect of Carbon Dose

To determine the influence of carbon dose, 25 mL of 20 mg/L solution at pH 5 were added with different amount of coconut coir activated carbon varies from 2 g/L to 10 g/L. The contact time used was 150 min and pH 5. From the observation of the Table 4.3 of the effect of carbon dose on adsorption of lead, the adsorption increased with the dose of carbon.

Table 4.3: Effect of carbon dose on adsorption of lead onto coconut coir activated carbon.

Carbon Dose (g/L)	Co	Ct	Ads (%)
2	20.0042	16.4328	17.8532
4	20.0042	12.1719	39.1532
5	20.0042	7.5483	62.2664
6	20.0042	3.4539	82.7341
8	20.0042	0.5439	97.2810
10	20.0042	0.5043	97.4790

The effect of carbon dose on removal of lead was summarized in figure below. From Figure 4.8, it is evident that the removal of lead increased with increasing carbon dose. A maximum removal of lead of 20 mg/L concentration was achieved with the carbon dose of 8 g/L. This result showed similar trend to lead removal by silica ceramic (Salim and Munekage, 2009); by coconut pith activated carbon (Santhy and Selvapathy, 2004); and by heartwood of areca catechu powder (Chakravarty *et al.*, 2010)

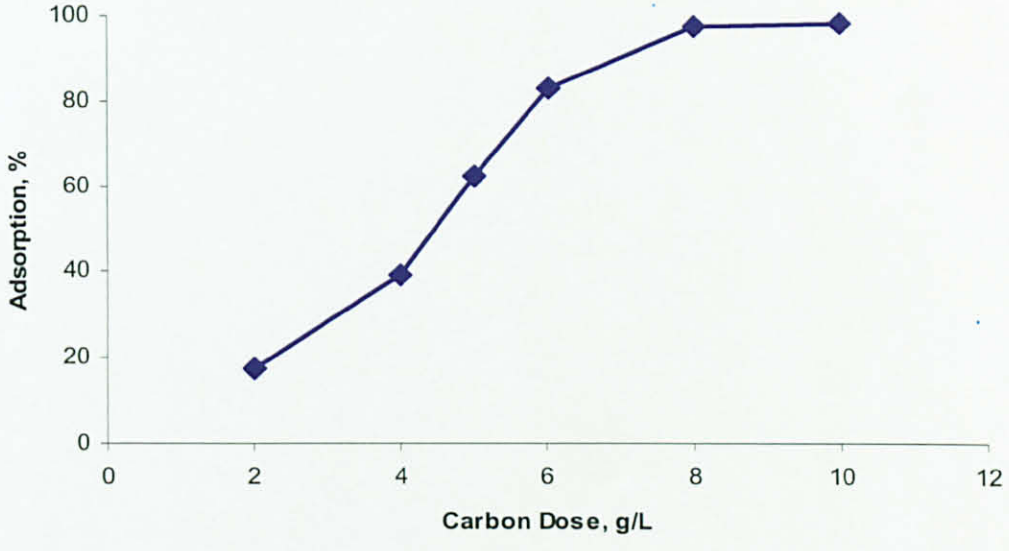

Figure 4.8: Effect of carbon dose on adsorption of lead onto coconut coir activated carbon.

4.3.2 Batch Study Using F-400 Activated Carbon

The efficiency of coconut coir activated carbon for adsorption of lead was compared to that of F-400 activated carbon. The adsorption rate of lead onto F-400 in batch adsorption system was done by investigating the effect of pH and contact time. The percentage removal of adsorbate uptake can be calculated by the same method mention before:

$$Adsorpbate\ uptake\ (\%) = (C_o - C_t) / C_o \times 100$$

where, C_o and C_t are the initial and at time (t) of the adsorbent concentration, respectively.

4.3.2.1 Effect of pH

To determine the influence of pH, 0.1 g of F-400 were added to series of flasks containing 25 mL of 20 mg/L solutions and contact time used was 24hr. The pH of solutions was varied from pH 1.0 to pH 6.0. From the experimental results, in Table 4.4 shows that sorption is very low at acidic pH and increases with increasing pH of lead solution. The optimum lead removal was observed at pH 5.5.

Table 4.4: Effect of pH on adsorption of lead onto F-400 activated carbon

pH	Co	Ct	Ads (%)
1	22.4274	21.5763	3.794911581
1.5	22.4274	21.4821	4.214933519
2	22.4274	21.0343	6.211598313
2.5	22.4274	19.93	11.13548606
3	22.4274	18.3836	18.03062326
3.5	22.4274	17.1028	23.74149478
4	22.4274	16.0321	28.51556578
4.5	22.4274	15.2892	31.82803178
5	22.4274	14.9987	33.12332236
5.5	22.4274	14.9902	33.16122243
6	22.4274	14.9897	33.16345185

The effect of pH on the removal of lead is summarized in Figure 4.9. From the Figure 4.10, it is evident that the adsorption increased with the increasing of pH and optimum pH is pH 5.5.

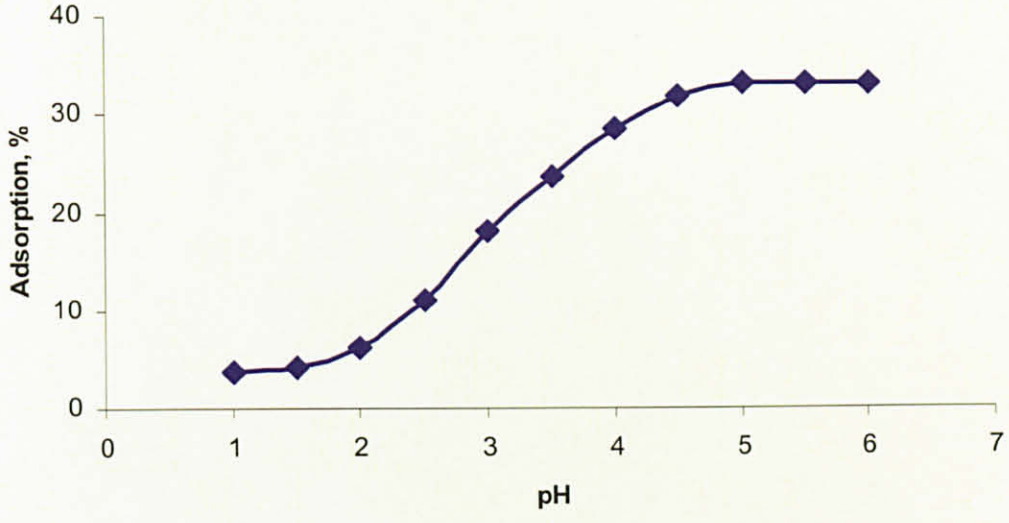

Figure 4.9: Effect of pH on adsorption of lead onto F-400 activated carbon

4.3.2.2 Effect of Contact Time

Effect of contact time on removal of Pb(II) is shown in Table 4.5. The contact time was varied from 0 to 300 min for concentration of 20 mg/L of lead solution at pH 5.5 in an orbital shaker. Removal of lead was found to be complete in 210 min.

Table 4.5: Effect of contact time on adsorption of lead onto F-400 activated carbon

Time (min)	Co	Ct	Ads (%)
0	19.92	19.6905	1.152108
10	19.92	18.8421	5.411145
20	19.92	17.4829	12.23444
30	19.92	16.352	17.91165
45	19.92	15.4328	22.5261
60	19.92	14.6872	26.26908
90	19.92	14.1138	29.14759
120	19.92	13.7847	30.7997
150	19.92	13.5614	31.92068
180	19.92	13.4593	32.43323
210	19.92	13.3057	33.20432
240	19.92	13.3053	33.20633
270	19.92	13.3058	33.20382
300	19.92	13.3056	33.20482

Figure 4.10 shows the effect of contact time on the adsorption of lead. Adsorption percentage increased with the increase of contact time and reached equilibrium at 210 min.

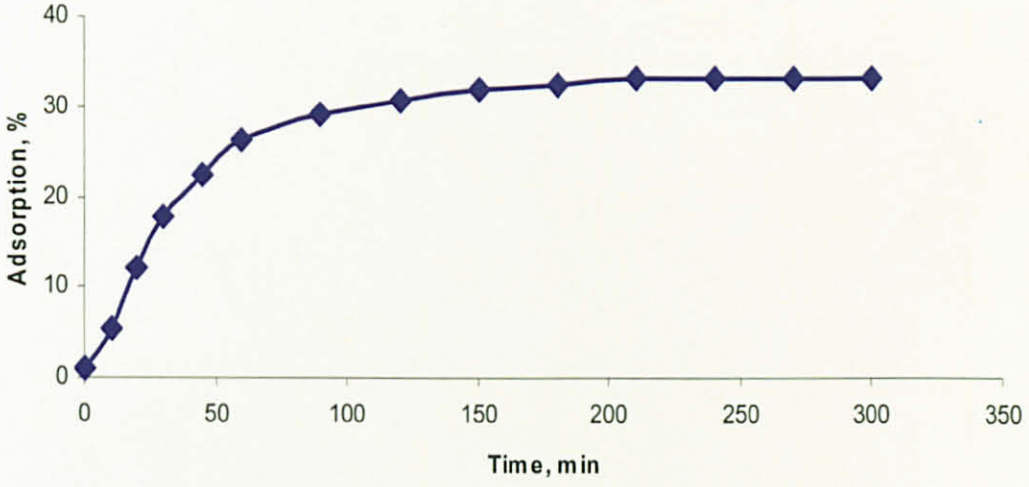

Figure 4.10: Effect of contact time on lead adsorption by F-400 activated carbon.

4.4 Adsorption Kinetic Study

Adsorption kinetic data of lead adsorption were fitted to Lagergren pseudo-first-order and pseudo-second-order kinetics.

4.4.1 Pseudo-First-Order Kinetics

Experimental kinetic data for lead adsorption are shown in Figure 4.11 where the fitted pseudo-first-order kinetics model best straight line is shown.

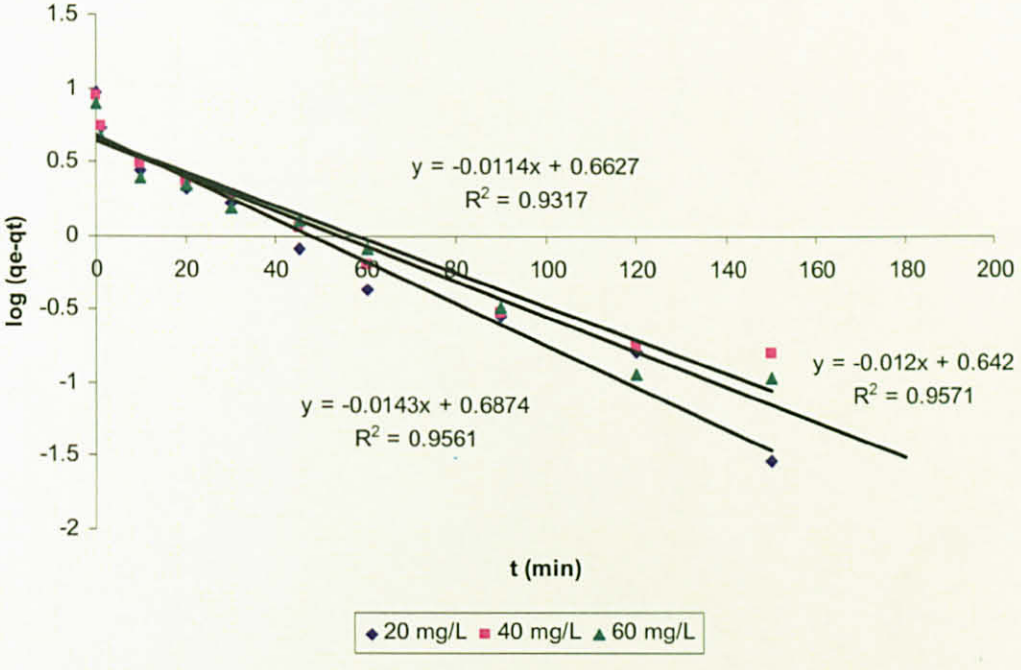

Figure 4.11: Pseudo-first-order kinetics plot for lead adsorption by coconut coir activated carbon

The kinetic parameters for the adsorption of lead onto coconut coir activated carbon are given in Table 4.6. The value of K_1 (slope) and q_e (y-intercept) indicating the rate constant of the adsorption and amount of lead ion adsorbed are shown in table.

Table 4.6: Pseudo-first-order kinetics model parameters for lead adsorption by coconut coir activated carbon

Lead Concentration	Pseudo-first-order		
	q_e	K_1	R^2
20 mg/L	4.87	0.033	0.9561
40 mg/L	4.59	0.026	0.9317
60 mg/L	4.38	0.027	0.9571

4.4.2 Pseudo-Second-Order Kinetics

Experimental kinetic data for lead adsorption are shown in Figure 4.12 where the fitted pseudo-second-order kinetics model best straight line is shown.

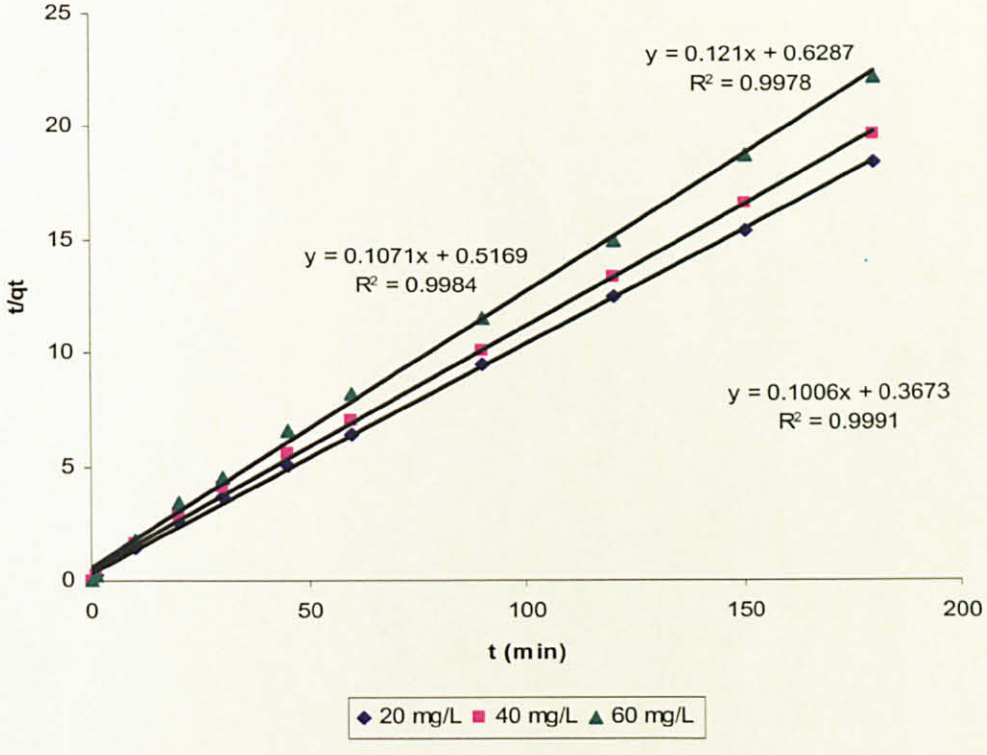

Figure 4.12: Pseudo-second-order kinetics plot for lead adsorption by coconut coir activated carbon

The kinetic parameters for the adsorption of lead onto coconut coir activated carbon are given in Table 4.7. The value of K_2 (y-intercept) and q_e (slope) indicating the rate constant of the adsorption and sorption capacity are shown in table.

Table 4.7: Pseudo-second-order kinetics model parameters for lead adsorption by coconut coir activated carbon

Lead Concentration	Pseudo-second-order		
	q_e	K_2	R^2
20 mg/L	9.94	0.027	0.9991
40 mg/L	9.34	0.022	0.9984
60 mg/L	8.26	0.023	0.9978

By comparing the constants of the two kinetic models, the Lagergren pseudo-second-order kinetics model shows the best fir for the experiment. Adsorption of lead from aqueous solution by coconut coir activated carbon follows pseudo-second-order kinetics which indicated that adsorption controlled by chemical sorption.

4.5 Adsorption Isotherm Study

Adsorption isotherm data of lead adsorption were fitted to well-known and widely applied isotherm models of Langmuir and Freundlich.

4.5.1 Freundlich Isotherm

Experimental isotherm data for lead adsorption are shown in Figure 4.13 where the fitted Freundlich isotherm best straight line is shown. This indicates that the adsorption process for both activated carbons follow the Freundlich model.

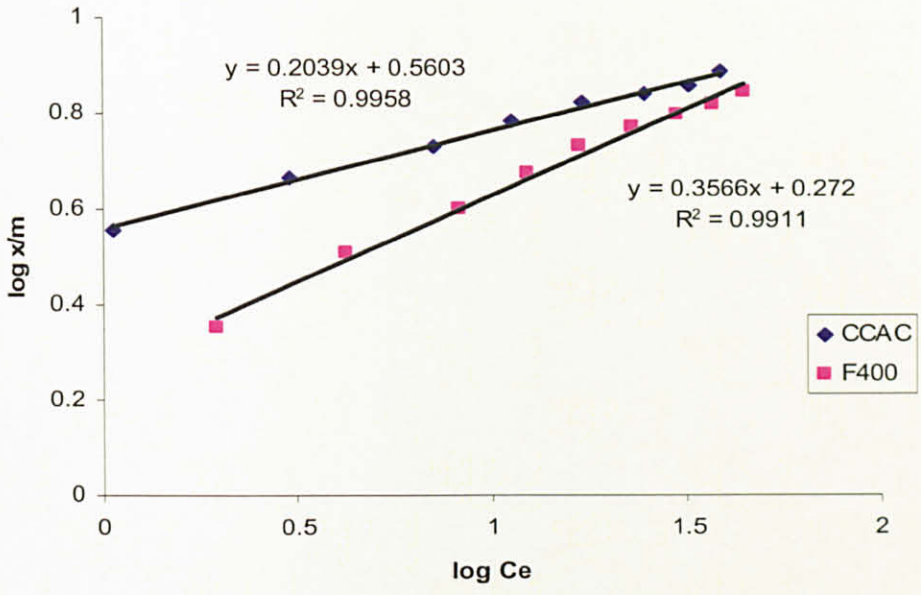

Figure 4.13: Freundlich isotherms for lead adsorption by coconut coir activated carbon and F-400 activated carbon.

The Freundlich isotherm parameters for the adsorption of lead onto coconut coir activated carbon and F-400 activated carbon are given in Table 4.8. The value of K_F (y-intercept) and $1/n$ (slope) indicating the adsorption capacity and adsorption intensity are shown in table.

Table 4.8: Freundlich isotherm constants

Activated Carbon	$1/n$	K_F	R^2
Coconut Coir	0.20	3.63	0.9958
F-400	0.36	1.87	0.9901

Conducting adsorption isotherm studies has shown that coconut coir has higher adsorption capacity compared to F-400 activated carbon. The adsorption intensity of F-400 is higher compared to that of coconut coir activated carbon. These results show that the coconut coir activated carbon is better compared to the F400 activated carbon.

4.5.2 Langmuir Isotherm

The same experimental adsorption data were fitted to the Langmuir Isotherm. The plotted graphs are shown in Figure 4.14.

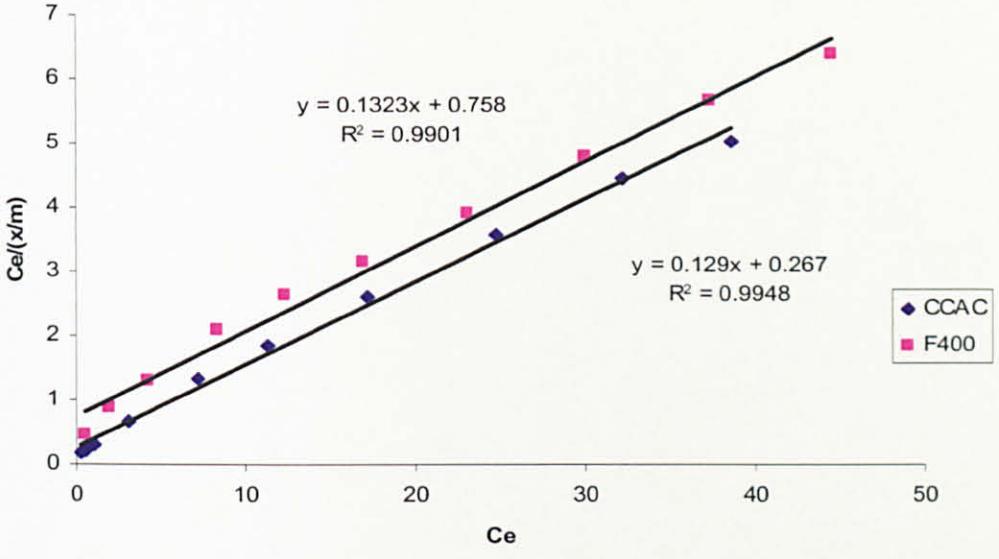

Figure 4.14: Langmuir isotherm for lead adsorption by coconut coir activated carbon and F-400 activated carbon.

Adsorption capacities and adsorption energy of both coconut coir activated carbon and F400 commercial activated for lead adsorption was determined by empirical constant $Q°$ and b of the Langmuir model. The empirical constants are summarized in Table 4.9.

Table 4.9: Langmuir isotherm constants

Activated Carbon	b	$Q°$	R^2
Coconut Coir	0.48	7.75	0.9948
F-400	0.17	7.55	0.9901

Langmuir Isotherm has shown that the coconut coir activated carbon has higher adsorption capacity and adsorption energy compared to F-400 activated carbon.

From both of these isotherms, it is shown that the adsorption data fitted well to both Freundlich and Langmuir isotherm for the adsorption of lead from aqueous solution onto coconut coir activated carbon and F-400 activated carbon.

4.6 Lead Adsorption Capacity of Different Adsorbents

The adsorption capacity of the adsorbents for the removal of lead was compared with those of other adsorbents reported in the literature and the values of adsorption capacities are presented in Table 4.10.

The experimental data of the present study are comparable with the reported values in some cases. From the table, it is shown that coconut coir activated carbon has a considerably high adsorption capacity of Langmuir and Freundlich constants.

Table 4.10: Lead adsorption capacity of different adsorbents

Adsorbent	pH	Temperature (°C)	Concentration Range (mg/L)	Langmuir Capacity ($Q°$)	Freundlich Capacity (K_F)	References
Bamboo dust carbon	7.2	30	100-1000	2.151	3.302	Kannan and Veemaraj (2009)
Periwinkle shell activated carbon	8.0	22	20-100	0.056	6.55	Badmus *et al.* (2007)
Silica ceramic	3.5	30	5-70	2.76	-	Salim M. and Munekage (2009)
Hazelnut husk activated carbon	5.7	22	10-200	13.05	2.13	Imamoglu *et al.* (2008)
Bael tree leaf powder	5	30	50-100	4.06	0.74	Kumar and Gayathri (2009)
Okra wastes	5.0	25	25-100	5.74	0.67	Hashem (2007)
Coconut coir activated carbon	5.0	22	10-100	7.75	3.63	This study
F-400 activated carbon	5.5	22	10-100	7.55	1.87	This study

CHAPTER 5
CONCLUSIONS

The results of this study show that the activated carbon prepared from coconut coir has suitable adsorption capacity for the removal of lead from aqueous solution. Adsorption increases with increase in contact time and carbon dose while decreases with the increase in lead concentration at optimum pH 5.

According to the Freundlich isotherm, coconut coir has higher adsorption capacity but lower adsorption intensity for lead adsorption compared to the F-400 activated carbon. However, according to Langmuir isotherm, coconut coir activated carbon exhibited higher adsorption capacity as well as the adsorption energy compared to the F-400 activated carbon.

Adsorption capacity of coconut coir activated carbon for lead is 7.75 (Langmuir) and 3.63 (Freundlich), whereas adsorption capacity of F-400 activated carbon is 7.55 (Langmuir) and 1.87 (Freundlich).

Coconut coir activated carbon is a suitable substitute for commercial activated carbon for adsorption of lead from aqueous solution.

CHAPTER 6
ECONOMICS BENEFIT

6.1 Cost of the Study

The materials, chemicals and equipment sources and estimated costs involved in the research are listed in the table below.

Table 6.1: Cost of the study

Material/Chemical/ Equipment	Quantity	Source	Cost
Coconut coir	1 kg	Taman Maju nursery	RM 0.80
Lead (II) Nitrate	100 g	Chemical Engineering Laboratory	-
Potassium Hydroxide (10%)	9 L	Chemical Engineering Laboratory	-
Hydrochloric Acid (0.01N)	100 ml	Civil Engineering Laboratory	-
Sodium Hydroxide (0.01N)	100 ml	Civil Engineering Laboratory	-
Fixed Bed Activation Unit	1	Chemical Engineering Laboratory	-
Atomic Adsorption Spectrophotometer (AAS)	1	Chemical Engineering Laboratory	-
Variable Pressure Scanning Electron Microscope (VPSEM)	1	Mechanical Engineering Laboratory	-
		Total Cost	**RM 0.80**

The cost identified in the study only in the raw material used to make coconut coir activated carbon needed for the experiments. Other chemicals and equipments needed for the experiments were provided by the laboratories in the campus. However, coconut coir is easily available since it is one of the agricultural byproducts in Malaysia, where people consume only the inner part of the coconut rather than making use of the skin. Thus, the cost for purchasing coconut coir can be avoided by getting the coconut coir waste from the villages or in the markets.

REFERENCES

Atkins P.W. 1994. *"Physical Chemistry"*, fifth edition, W.H. Freeman and Corporation New York, New York.

Badmus M.A.O., Audu T.O.K. and Anyata B.U. 2007. *"Removal of Lead Ion from Industrial Wastewater by Activated Carbon Prepared from Periwinkle Shells (Typanotonus Fuscatus)"*, Turkish Journal of Engineering, Environmental and Science, 31, 251-263.

Burchell T.D. 1999. *"Carbon Materials for Advanced Technologies"*, first edition, Elsevier Science Ltd.

Chakravarty P., Sarma N.S. and Sarma H.P. 2010. *"Removal of Lead(II) from Aqueous Solution using Heartwood of Areca Catechu Powder"*, Desalination, 256, 16-21.

El-ashtoukhy E.-S.Z., Amin N.K. and Abdelwahab O. 2008. *" Removal of Lead(II) and Copper(II) from Aqueous Solution using Pomegranate Peel as a New Adsorbent"*, Desalination, 223, 162-173.

Esalah J.O., Weber M.E. and Vera J.H. 1999. *"Removal of Lead from Aqueous Solution by Precipitation with Sodium Di-(n-octyl) Phosphinate"*, Separation and Purification Technology, 18, 25-36.

Febrianto J., Kosasih A.N., Sunarso J., Ju Y.H., Indraswati N. and Ismadji S. 2009. *"Equilibrium and Kinetic Studies in Adsorption of Heavy Metals using Biosorbent: A Summary of Recent Studies"*, Journal of Hazardous Material, 162, 616-645.

Gimba C.E., Turoti M., Egwaikhide P.A. and Akporhonor E.E. 2009. *"Adsorption of Indigo Blue Dye and Some Toxic Metals by Activated Carbons from Coconut Shells"*, Electronic Journal of Environmental, Agricultural and Food Chemistry, 8, 1194-1201.

Hashem M.A. 2007. *"Adsorption of Lead Ions from Aqueous Solution by Okra Wastes"*, International Journal of Physical Sciences, 2, 178-184.

Imamoglu M. and Tekir O. 2008. *"Removal of Copper(II) and Lead(II) Ions from Aqueous Solutions by Adsorption on Activated Carbon from a New Precursor Hazelnut Husks"*, Desalination, 228, 108-113.

Kadirvelu K., Thamaraiselvi K. and Namasivayam C. 2001. *"Removal of Heavy Metals from Industrial Wastewaters by Adsorption onto Activated Carbon Prepared from an Agricultural Solid Waste"*, Bioresource Technology, 76, 63-65.

Kannan N. and Veemaraj T. 2009. *"Removal of Lead(II) Ions by Adsorption onto Bamboo Dust and Commercial Activated Carbons-A Comparative Study"*, E-Journal of Chemistry, 6(2), 247-256.

Kumar P.S. and Gayathri R. 2009. *"Adsorption of Pb^{2+} Ions from Aqueous Solutions onto Bael Tree Leaf Powder: Isotherms, Kinetics and Thermodynamics Study"*, Journal of Science and Technology, 4, 381-399.

Lee M.Y., Lee S.H., Shin H.J., Kajiuchi T. and Yang J.W. 1998. *"Characteristics of Lead Removal by Crab Shell Particles"*, Process Biochemistry, 33, 749-753.

Liu Z. and Zhang F.S. 2009. *"Removal of Lead from Water using Biochars Prepared from Hydrothermal Liquefaction of Biomass"*, Journal of Hazardous Material, 167, 933-939.

Marsh H. and Rodriguez F. 2006. *"Activated Carbon"*, first edition, Elsevier Science Ltd.

Metcalf and Eddy. 2004. *"Wastewater Engineering Treatment and Reuse"*, fourth edition, McGraw Hill.

Mouflih M., Aklil A., Jahroud N., Gourai M. and Sebti S. 2005. *"Removal of Lead from Aqueous Solution by Natural Phosphate"*, Hydrometallurgy, 81, 219-225.

Noroozi B., Sorial G.A., Bahrami H. and Arami M. 2008. *"Adsorption of Binary Mixtures of Cationic Dyes"*, Dyes and Pigments, 76, 784-791.

Quek S.Y., Al-Duri B., Wase D.A.J. and Forster C.F. 1998. *"Coir as Biosorbent of Copper and Lead"*, Institution of Chemical Engineers, 76, 50-54.

Reed B.E. 2002. *"Removal of Heavy Metals by Activated Carbon"*, Lewis Publishers.

Salim M. and Munekage Y. 2009. *"Lead Removal from Aqueous Solution using Silica Ceramic: Adsorption Kinetics and Equilibrium Studies"*, International Journal of Chemistry, 1, 1.

Santhy K. and Selvapathy P. 2004. *"Removal of Heavy Metals from Wastewater by Adsorption on Coir Pith Activated Carbon"*, Separation Science and Technology, 39, 3331-3351.

Site A.D. 2001. *"Factors Affecting Sorption of Organic Compounds in Natural Sorbent/Water Systems and Sorption Coefficients for Selected Pollutants"*, Journal of Physical and Chemical Reference Data, 30, 187-439.

Tangjuank S., Insuk N., Tontrakoon J. and Udeye V. 2009. *"Adsorption of Lead(II) and Cadmium(II) Ions from Aqueous Solutions by Adsorption on Activated Carbon Prepared from Cashew Nut Shells"*, World Academy of Science, Engineering and Technology, 52.

Tchobanoglous G., Burton F.L. and Stensel H.D. 2004. *"Wastewater Engineering: Treatment and Reuse"*, McGraw Hill, New York.

Weber W.J. 1972. *"Physicochemical Processes for Water Quality Control"*, Wiley Interscience, New York.

Weber W.J. and Morris J.C. 1963. *"Kinetics of Adsorption on Carbon from Solution"*, Journal of Science Interface, 89, 31-59.

Xu T. and Liu X. 2008. *"Peanut Shell Activated Carbon: Characterization, Surface Modification and Adsorption of Pb^{2+} from Aqueous Solution"*, Chineese Journal of Chemical Engineering, 16, 401-406.

Appendix A: Adsorption Isotherm Using Coconut Coir Activated Carbon

Co	Ce	Adsorption (%)	Co-Ce	(Co-Ce)/Co	V	(Co-Ce)V	m	x/m	Ce/(x/m)	log Ce	log (x/m)
10	0.2395	97.605	9.7605	0.9760	0.1	0.9760	0.8	1.2200	0.19630	-0.62069	0.08638
20	0.5423	97.2885	19.4577	0.9728	0.1	1.9457	0.8	2.4322	0.2229	-0.26576	0.38600
30	1.0522	96.4926	28.9478	0.9649	0.1	2.8947	0.8	3.6184	0.2907	0.02209	0.55852
40	3.0133	92.4667	36.9867	0.9246	0.1	3.6986	0.8	4.6233	0.6517	0.47904	0.66495
50	7.1246	85.7508	42.8754	0.8575	0.1	4.2875	0.8	5.3594	1.3293	0.85276	0.72911
60	11.3167	81.1388	48.6833	0.8113	0.1	4.8683	0.8	6.0854	1.8596	1.05371	0.78429
70	17.1967	75.4331	52.8032	0.7543	0.1	5.2803	0.8	6.6004	2.6054	1.23544	0.81957
80	24.6878	69.1402	55.3122	0.6914	0.1	5.5312	0.8	6.9140	3.5706	1.39248	0.83973
90	32.2324	64.1862	57.7676	0.6418	0.1	5.7767	0.8	7.2209	4.4637	1.50829	0.85859
100	38.5451	61.4549	61.4549	0.6145	0.1	6.1454	0.8	7.6818	5.0176	1.58596	0.88546

Appendix B: Adsorption Isotherm Using F400 Activated Carbon

Co	Ce	Adsorption (%)	Co-Ce	(Co-Ce)/Co	V	(Co-Ce)V	m	x/m	Ce/(x/m)	log Ce	log (x/m)
10	0.5334	94.6660	9.4666	0.9466	0.1	0.94666	0.8	1.18332	0.45076	-0.27294	0.07310
20	1.9563	90.2185	18.0437	0.9021	0.1	1.80437	0.8	2.25546	0.86736	0.29143	0.35323
30	4.1987	86.0043	25.8013	0.8600	0.1	2.58013	0.8	3.22516	1.30185	0.62311	0.50855
40	8.2719	79.3200	31.7280	0.7932	0.1	3.17280	0.8	3.96600	2.08571	0.91760	0.59835
50	12.3535	75.2930	37.6465	0.7529	0.1	3.76465	0.8	4.70581	2.62515	1.09179	0.67263
60	16.9649	71.7251	43.0350	0.7172	0.1	4.30350	0.8	5.37938	3.15368	1.22955	0.73073
70	23.0362	67.0911	46.9638	0.6709	0.1	4.69638	0.8	5.87047	3.92407	1.36241	0.76867
80	29.9517	62.5603	50.0483	0.6256	0.1	5.00483	0.8	6.25603	4.78764	1.47642	0.79629
90	37.2626	58.5970	52.7373	0.5859	0.1	5.27373	0.8	6.59217	5.65256	1.57127	0.81902
100	44.4723	55.5277	55.5277	0.5552	0.1	5.55277	0.8	6.94096	6.40722	1.64808	0.84141